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IS 1698 (1994): Indigo Carmine, Food Grade [FAD 8: Food Additives]

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**IS 1698 : 1994**
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# इंडिगो कारमिन, खाद्य ग्रेड — विशिष्टि

( दूसरा पुनरीक्षण )

*Indian Standard*

# INDIGO CARMINE, FOOD GRADE — SPECIFICATION

*( Second Revision )*

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First Reprint JUNE 2001

UDC 664.099.62 : 667.286.2

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*November* 1994

**Price Group 2**

# AMENDMENT NO.1 MAY 2008
# TO
# IS 1698 : 1994 INDIGO CARMINE, FOOD GRADE — SPECIFICATION

***( Second Revision )***

[*Page* 1, *Table* 1, *Sl No.* (iii), *col* 3] — Substitute '0.2' *for* '0.4'.

[*Page* 1, *Table* 1, *Sl No.* (iv), *col* 3] — Substitute '0.2' *for* '0.4'.

[*Page* 1, *Table* 1, *Sl No.* (v), *col* 3] — Substitute '1.0' *for* '3.0'.

[*Page* 1, *Table* 1, *Sl No.* (vi), *col* 3] — Substitute '0.5' *for* '1'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

Food Additives Sectional Committee, FAD 8

## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agricultural Division Council.

This standard is one of the series of Indian Standards for edible synthetic colours permitted under the *Prevention of Food Adulteration Rules*, 1955 issued by the Ministry of Health, Government of India. These rules, *inter-alia* prescribe:

> 'All food colours including natural colouring matter and permitted synthetic food colours and their preparations or mixtures excluding saffron and curcumin shall be sold only under the BIS Certification Mark.'

'Indian Standard specification for indigo carmine' was first issued in 1960. It was revised in 1974 to bring it in line with the FAO/WHO specifications. This standard is being revised again taking into consideration the latest publication for the food colour issued by FAO/WHO [*see* FAO Food and Nutrition Paper 31/1(1984) Specification for identity and purity of food colours], the latest specifications laid down under Food Chemical Codex, EEC Directives, Canadian Food Laws, Food and Drugs Act of USA. In addition, due consideration has been given to indigenous data.

In this revision the limits for water insoluble matter, combined ether extracts and subsidiary dyes have been made more stringent to align them with the International requirements and requirements for heavy metals have been included.

Requirements for indigo carmine, food grade have been prescribed under the *Prevention of Food Adulteration Rules*, 1955. As food colours are under compulsory BIS Certification, the modified values given in Table 1 have been referred to CCFS for adoption under the PFA Rules. While formulating this standard, necessary consideration has been given to the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977. The standard is however subject to restrictions imposed under these Rules, wherever applicable.

**Description**

*Common Name* — Indigo carmine

*Synonyms* — Indigotine, FD & C Blue No. 2, C.I. Food Blue 1, E.E.C. Serial No. E 132, L Blau 2

*Colour of the 0.1 Percent (m/v) Solution in Distilled Water* — Blue

*Colour Index Number (1975)* — No. 73015

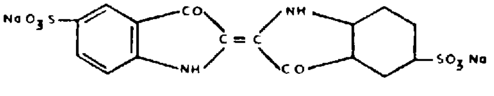
*Class* — Indigoid

*Chemical Name* — Disodium salt of indigotine 5,5'-disulphonic acid

*Empirical Formula* — $C_{16}H_8N_2O_8S_2Na_2$

*Molecular Weight* — 466.36

*Structural Formula*



*Solubility* — Soluble in water

Sparingly soluble in ethanol

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

# INDIGO CARMINE, FOOD GRADE — SPECIFICATION

## *( Second Revision )*

## 1 SCOPE

**1.1** This standard prescribes the requirements and the methods of sampling and test for indigo carmine, food grade.

## 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| 1070 : 1992 | Reagent grade water (*third revision*) |
| 1699 : 1994 | Methods of sampling and test for food colours (*second revision*) |
| 2491 : 1972 | Code for hygienic conditions for food processing units (*first revision*) |

## 3 REQUIREMENTS

**3.1** The material shall conform to the requirements prescribed in Table 1.

### 3.2 Freedom from Contaminants

Precautions shall be taken to ensure that the material is free from mercury, copper and chromium in any form; aromatic amines, aromatic nitro compounds, aromatic hydrocarbons, and cyanides.

**Table 1 Requirements for Indigo Carmine**

( *Clause* 3.1 )

| Sl No. | Characteristic | Requirement | Method of Test, Ref to | |
|---|---|---|---|---|
| | | | Annex of This Standard | Clause of IS 1699 : 1994 |
| (1) | (2) | (3) | (4) | (5) |
| i) | Total dye content, corrected for sample dried at 105 ± 1°C for 2 h, percent by mass, *Min* | 85 | A | — |
| ii) | Loss on drying at 135°C, percent by mass | 15 | — | 6 |
| | and | | | |
| | Chloride and sulphates expressed as sodium salt, percent by mass, *Max* | | | 13<br>14 |
| iii) | Water-insoluble matter, percent by mass, *Max* | 0.4 | B | — |
| iv) | Combined ether extracts, percent by mass, *Max* | 0.4 | C | — |
| v) | Subsidiary dyes, percent by mass, *Max* | 3.0 | D | — |
| vi) | Isatin sulphonic acid, percent by mass, *Max* | 1 | E | — |
| vii) | Lead, mg/kg, *Max* | 10 | — | 15 |
| viii) | Arsenic, mg/kg, *Max* | 3 (*see* Note) | — | 15 |
| ix) | Heavy metals, mg/kg, *Max* | 40 | — | 16 |

NOTE — The value of arsenic has been referred to CCFS for adoption under the PFA Rules.

**3.3** The product shall be processed, packed, stored and distributed under hygienic conditions in licenced premises (*see* IS 2491 : 1972).

## 4 PACKING AND MARKING

### 4.1 Packing

The material shall be packed in glass containers, polyethylene container, metal containers, or cardboard containers suitably lined with polyethylene. Subject to agreement between the purchaser and the vendor any other suitable container may also be used.

### 4.2 Marking

**4.2.1** Each container shall be legibly and indelibly marked with the following information:

a) The words '*FOOD COLOUR*';
b) Common name of the colour;
c) Chemical name of the colour;
d) Colour index number;
e) Source of manufacture;
f) Date of manufacture;
g) Net mass in grams or kilograms;
h) Batch or code number; and
j) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules,* 1955.

**4.2.2** *BIS Certification Marking*

The product may also be marked with the Standard Mark.

**4.2.2.1** The use of the Standard Mark is governed by the Provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 5 SAMPLING

**5.1** Representative samples of the material for tests shall be drawn and criteria for ascertaining conformity to the requirements of this specification shall be determined according to the method prescribed in 4 of IS 1699 : 1994.

## 6 TESTS

**6.1** Tests shall be carried out as prescribed in col 4 and 5 of Table 1.

### 6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## ANNEX A

[ *Table* 1, *Item* (i) ]

## DETERMINATION OF TOTAL DYE CONTENT

### A-0 GENERAL

Two methods, spectrophotometric method and the titanium trichloride method, are specified. In case of dispute, spectrophotometric method shall be regarded as the reference method.

### A-1 SPECTROPHOTOMETRIC METHOD

### A-1.1 Apparatus

Suitable spectrophotometer with properly calibrated scales for both wavelength and optical density. However, a suitable spectrocolorimetric may also be used after calibration against a spectrophotometer.

### A-1.2 Reagents

**A-1.2.1** *Ammonium Acetate Solution*

200 mg of ammonium acetate in one litre of water (re-distilled).

### A-1.3 Procedure

Weigh accurately about 250 mg of the dye sample and dissolve with ammonium acetate solution in a 250-ml volumetric flask; dilute this with the same solvent to make a final concentration of 1 mg/100 ml (approximately). Find out the optical density of this dilute solution, against ammonium acetate solution as blank, at 610 nm, in a glass cell with 10.0 mm light path.

**A-1.3.1** Simultaneously weigh accurately about 2 g of the dye-sample and dry this in an air-oven at 105 ± 1°C for 2 hours. Calculate the loss of mass on drying; from this data, calculate the dry mass of the sample ($M$) in the final solution taken for measurement of optical density.

### A-1.4 Calculation

$$\text{Total dye content in the sample, percent} = \frac{OD \times 100}{M \times 450}$$

where

*OD* = optical density found;

*M* = dry mass of the sample in 100 ml solution; and

450 = $E^{1\%}$1cm, 610 nm, for indigo carmine in ammonium acetate solution.

**A-2 TITANIUM TRICHLORIDE METHOD**

**A-2.1 Procedure**

The method given in 5 of IS 1699 : 1994 shall be followed. The percentage of total dye content shall be determined using the following calculations:

1 ml of 0.1 N $TiCl_3$ = 0.023 32 g of indigo carmine.

## ANNEX B

[*Table* 1, *Item* (iii)]

### DETERMINATION OF WATER INSOLUBLE MATTER

**B-1 PROCEDURE**

The method given in **7** of IS 1699 : 1994 shall be followed taking 2 gram of sample instead of 4.5-5 g. (*see* **7.2** of IS 1699 : 1994).

## ANNEX C

[*Table* 1, *Item* (iv)]

### DETERMINATION OF COMBINED ETHER EXTRACT

**C-1 PROCEDURE**

The method given in **8** of IS 1699 : 1994 shall be followed taking 2 g of sample instead of 5 g (*see* **8.4** of IS 1699 : 1994).

## ANNEX D

[*Table 1, Item (v)*]

### DETERMINATION OF SUBSIDIARY DYE CONTENT

**D-1 PROCEDURE**

The method given in **9** of IS 1699 : 1994 using the conditions given below shall be followed:

Developing solvent: No. 3

Height of ascent of solvent front : approximate 17 cm

NOTES

**1** The 5,7 isomer is separated as a wide blue zone just in front of the main blue band. Do not include this zone in the subsidiary colouring matter zones which are cut out and measured.

**2** The 15 ml sodium hydrogen carbonate solution used in the general procedure is replaced by 15 ml 0.05 *N* hydrochloric acid in order to avoid the decomposition which sulfonated indigo undergoes in alkaline solution

## ANNEX E

[*Table* 1, *Item* (vi)]

### DETERMINATION OF THE ISATIN SULPHONIC ACID

**E-1 PRINCIPLE**

**E-1.1** Separate isatin sulphonic acid from indigo carmine by ascending paper chromatography and extract it from the paper. Measure the optical density of the extract at the wavelength of maximum absorbance around 247 nm, from which calculate the content of the intermediate as a percent by mass of the sample.

**E-2 APPARATUS**

**E-2.1 Chromatograph Tank with Ancillary Equipment** — As given under **7.2.1** of IS 1699 : 1974.

**E-2.2 Microsyringe** — Capable of delivering 0.2 ml with a tolerance of ± 0.002 ml.

**E-2.3 Spectrophotometer**

**E-3 REAGENTS**

**E-3.1 Atmosphere Saturating Solvent** — Water.

**E-3.2 Developing Solvent** — Sodium chloride solution 15 percent.

**E-3.3 Extracting Solvent** — Ammonium sulphate solution, 25 percent.

## E-4 PROCEDURE

**E-4.1** Mark out a sheet of chromatography paper as shown in Fig. 2 of IS 1699 : 1994. Apply 0.2 ml of a 1.0 percent *m/v* (based on 100 percent purity) aqueous solution of the dye as uniformly as possible within the confines of the 180 mm × 7 mm rectangle, holding the nozzle of the micro-syringe steadily in contact with the paper. Allow the paper to dry at room temperature for 1 to 2 hours, or at 50°C for 5 minutes followed by 15 minutes at room temperature. Mount the sheet, together with a plain sheet to act as a blank, in frame *(C)*. Pour sufficient developing solvent into the tray *(D)* to bring the surface of the solvent about 1 cm below the base line of the chromatogram sheet. Put the frame *(C)* in position and replace the cover. Allow the solvent to ascent to the full height of the papers. Transfer the papers to a drying cabinet and dry at 50-60°C. Remove the sheets from frame *(C)*.

**E-4.2** Holding the chromatogram under ultraviolet light, mark the top and the bottom of the fluorescent band. (Rf value of isatin-5-sulphonic acid is 0.9). Cut the band from the sheet as a strip and cut an equivalent strip from the plain sheet. Extract each strip separately with 10 ml of the ammonium-sulphate solution and filter. Measure the optical density of the extracts at the peak near 247 ± 5 nm.

## E-5 CALCULATION

**E-5.1** Isatin sulphonic acid, $= (D-d) \times F$ percent by mass

where,

$D$ = the optical density of the isatin sulphonic acid extract,

$d$ = the optical density of the corresponding blank, and

$F$ = 6.0.

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This Indian Standard has been developed from Doc: No. FAD 8 ( 253 ).

**Amendments Issued Since Publication**

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
| | | |
| | | |

BUREAU OF INDIAN STANDARDS

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Printed by Reprography Unit, BIS, New Delhi